

Title	Spectrophotometric Method for Determination of Iron with Dibenzoylmethane
Author(s)	Shigematsu, Tsunenobu; Tabushi, Masayuki
Citation	Bulletin of the Institute for Chemical Research, Kyoto University (1961), 39(3): 260-261
Issue Date	1961-07-31
URL	http://hdl.handle.net/2433/75812
Right	
Type	Departmental Bulletin Paper
Textversion	publisher

ABSTRACTS

energy spectrum as in aqueous solution (fluorescence band: 540–700 $m\mu$, peak: 593 $m\mu$), and therefore a red filter which has the maximum transmission near 600 $m\mu$ is conveniently used as a secondary filter. Analytical procedure is as follows: To the sample solution, add 0.2 ml of 0.1% Pontachrome Blue Black R ethanol Solution and 5 ml of 10% ammonium acetate solution, dilute to about 50 ml with water and then adjust pH to 4.8. Heat in boiling water for 10 minutes, cool down to room temperature extract the complex formed with 25 ml of amyl alcohol, and measure the intensity of fluorescence of the organic layer.

By the procedure 0.05–1.5 μg of aluminium can be determined. Copper, cobalt and titanium give negative error while gallium a large positive one. The effects of ferric iron and vanadate can be prevented by reducing with hydroxylamine.

The method is suitable for the micro-determination of aluminium because of its high sensitivity. Micro amount of aluminium (0.004–0.012%) in pure magnesium metal could be determined and good results were obtained.

Spectrophotometric Method for Determination of Iron with Dibenzoylmethane

Tsunenobu SHIGEMATSU and Masayuki TABUSHI

*Nippon Kagaku Zasshi (Journal of the Chemical Society of
Japan, Pure Chemistry Section)*, 81, 262 (1960)

A spectrophotometric method for the determination of ferric iron based on the extraction of ferric-dibenzoylmethane chelate with butyl acetate was developed.

The pure crystalline powder of ferric dibenzoylmethane chelate was obtained, and the formula was estimated as $Fe[(C_6H_5CO)_2CH]_3$ from the iron content, 7.63% (theoretical 7.7%).

Ferric dibenzoylmethane chelate is soluble in organic solvents such as chloroform and butyl acetate. Absorption spectra of the chelate measured in chloroform and butyl acetate show two absorption bands in near ultraviolet region. The spectra of the chloroform solution, however, are unstable unless an excess of the reagent is present and are also affected considerably by the concentrations of the reagent. Therefore, the spectra of the chloroform solution are not suitable for the determination of iron. The chelate in butyl acetate medium shows absorption maxima at 320 $m\mu$ and 410 $m\mu$, the former of which, however, can not be used for the determination because of the intense absorption band of dibenzoylmethane.

In the region near 410 $m\mu$, the absorption spectra of the chelate extracted by the following procedure are the same as in the case above. Therefore the measurement of absorbances was made at 410 $m\mu$. The effects of the reagent concentrations and pH values were investigated, and the analytical procedure was established as follows: To the sample solution with pH 2.5–3.5, 0.5 ml. of 5% dibenzoylmethane acetone solution is added and the solution is warmed in a water bath at 70°C for 10 minutes. The chelate formed is extracted with 20 ml. of butyl acetate, and the

ABSTRACTS

absorbance of the organic layer is measured at $410\text{m}\mu$ against the reagent blank. Beer's law is obeyed up to 2 ppm. and molar extinction coefficient is $17,00\text{ l./mol. cm.}$ Molybdenum, titanium, copper and a large amount of chromium (III) interfere the determination of iron.

Separation and Spectrophotometric Determination of Uranium by the Extraction of the Dibenzoylmethane Chelate with Butyl Acetate

Tsunenobu SHIGMATSU and Masayuki TABUSHI

Nippon Kagaku Zasshi (Journal of the Chemical Society of Japan, Pure Chemistry Section), 81, 265 (1960)

Solvent extraction and spectrophotometric determination of uranium as a chelate with dibenzoylmethane were investigated. Dibenzoylmethane reacts with uranium to form a stable chelate, $\text{UO}_2[(\text{C}_6\text{H}_5\text{CO})_2\text{CH}]_2$, which is insoluble in water but soluble in chloroform or butyl acetate. The chloroform solution, however, is unstable as seen in the case of uranium acetylacetonate or ferric dibenzoylmethane chelate.

Absorption spectra of the uranium dibenzoylmethane chelate show three maxima at 275, 335 and $400\text{m}\mu$. The last maximum can be used for the determination of uranium.

The effects of the reagent concentrations and pH values were investigated, and EDTA was introduced as a masking agent for the various cations. The analytical procedure was established as follows:

To the sample solution, 2 ml of 10% EDTA solution and calcium chloride solution equivalent to EDTA are added, pH is adjusted to 6-7, and 0.5-1 ml of 5% dibenzoylmethane acetone solution is added. After diluting to about 40 ml, the solution is warmed at $60-70^\circ\text{C}$ for 10-15 minutes. The chelate formed is extracted with 20 ml of butyl acetate and the absorbance of organic layer is measured at $400\text{ m}\mu$ or $410\text{ m}\mu$ or $410\text{m}\mu$ against the reagent blank.

Beer's law is obeyed up to 10 ppm. and the molar extinction coefficients are $20,300\text{ l./mol. cm.}$ at $400\text{m}\mu$, and $17,680\text{ l./mol. cm.}$ at $410\text{m}\mu$. Copper gives a positive error and a large amount of titanium, chromium (III). and ferric irons interfere the determinations by the hydroxides precipitations.

In the procedure, the extraction recovery of uranium is quantitative and the extraction method is successfully applied to the separation of uranium from mixed fission products. The decontamination factor, 10^4 is given for a single extraction.